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ADVANCES IN THE DESIGN OF TOUGHENED CERAMICS

Paul F. Becher

Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee, USA

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The mechanical performance of brittle ceramics can be improved by increasing their fracture toughness/resistance. This is reflected by increases in resistance to strength degradation from in-service thermal stresses, slow crack growth, and contact or impact damage when the fracture toughness is enhanced. Furthermore, it appears that the distribution of the fracture strengths is narrowed (e.g., increased Weibull modulus) in toughened ceramics. Two approaches which can result in improved fracture toughness are transformation toughening and reinforcement processes. The reinforcement process can be achieved by introducing various microstructural features (e.g., strong whiskers and platelets, large noncubic matrix grains, and elongated matrix grains). However, the design of toughened ceramics employing either transformation toughening or reinforcement mechanisms must consider the material properties and characteristics which control each mechanism. Both the analysis of the toughening contributions and experimental observations can be used to define the influence of microstructure and material parameters on the toughening effects. These observations, then, provide a basis for the design of toughened ceramics.

Introduction

While very high fracture strengths (i.e, 700 to 1500 MPa) can be obtained in ceramics at moderate temperatures, this requires advanced processing and surface finishing techniques to achieve defect and flaw sizes which are less than 10 μm. This is a direct result of the inherent brittleness of ceramics which typically exhibit fracture resistances (toughnesses) of ≤ 5 MPa $m^{1/2}$ [1]. Even when high fracture strengths are achieved, the distribution of flaw and defect sizes typically lead to strengths which vary significantly. This is seen in low Weibull modulus values from the analysis of the probability of failure vs stress of large groups of specimen. Typically, Weibull modulus values of ≤ 6 are achieved in monolithic ceramics with toughnesses ≤ 5 MPa $m^{1/2}$. This means that even though the average fracture strength may be high (e.g., 700 MPa), a significant number will have strengths which are only a fraction of this. One can proof test components to eliminate those with strengths less than a desired level, but this is, of course, an expensive approach. More practical approaches include the development of processing techniques which minimize the number and size

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of defects introduced during various fabrication steps [2] and the design of materials with greater fracture resistance and, hence, less flaw sensitivity.

In fact, many applications of ceramics do not depend on the achievement of extremely high fracture strengths when subjected to flexural and tensile loads. The critical factors in the fracture strength of components are the attainment of strengths that are predictable and which are not degraded in service. The growth of existing flaws and the generation of new flaws due to slow crack growth, static and cyclic fatigue, thermal shock, impact and contact damage, and creep will result in a loss of strength. Except for creep resistance, the contribution of each of these factors is directly related to the fracture resistance or toughness of the ceramic. Therefore, it is imperative that approaches to increase the toughness be developed and incorporated into the design of the ceramic for a particular application.

Over the past decade or so, considerable advances have been made to improve the fracture toughness of ceramic systems. One of the most vivid examples is the toughening contribution from the stress-induced martensitic transformation of tetragonal zirconia grains [3],[4],[5]. Measurements in the author's laboratory show that the transformation toughening process can result in fracture toughness values of 10 to nearly 20 MPa $m^{1/2}$ in partially stabilized zirconias (PSZ) and tetragonal zirconia polycrystals (TZP) and 8 to ~15 MPa $m^{1/2}$ in zirconia toughened ceramics (ZTC) materials [6]. Other important advances in the toughening of ceramics include those related to crack pinning and deflection, crack bridging, and pullout by matrix grains, dispersed particles and reinforcing phases, and/or stress-induced microcracking [7],[8],[9],[10],[11]. These processes can be utilized to develop ceramics with toughnesses of upwards of 10 to 15 MPa $m^{1/2}$ in systems reinforced with discontinuous phases (e.g., whiskers, platelets, large and elongated grains,[6] and values in excess of 15 MPa $m^{1/2}$ using continuous fiber reinforcement) [12].

As shown in earlier studies, the incorporation of both transformation toughening and whisker reinforcement can be utilized not only to increase fracture toughness but also to increase the ceramic's resistance to thermal shock [13],[14], and slow crack growth under static [15],[16],[17] and cyclic [18] loading conditions. These same toughening mechanisms contribute to the achievement of narrow strength distributions (high Weibull moduli) as well [19],[20],[21]. As discussed by Kendall [22], the improved strength distributions can be rationalized in terms of the R-curve (increasing fracture resistance with crack extension) behavior which is observed in these same toughened ceramics. The conclusion of all these observations is that processes which enhance the fracture toughness of ceramics can and do result in major improvements in the overall mechanical performance of ceramics. Thus, it is imperative that design of the materials include factors contributing to improved fracture resistance such as the tailoring of the microstructure, the introduction of second phases which act as reinforcement or those that contribute transformation toughening, or combinations of these features.

In this paper, we consider the potential for raising the fracture resistance of ceramics by the incorporation of both zirconia phases which undergo a stress-induced martensitic phase transformation and strong, discontinuous, brittle reinforcing phases. In the case of ceramics containing tetragonal zirconia, the transformation-toughening contribution will be discussed in terms of the microstructural and compositional features which influence the stress-induced martensitic transformation. For ceramic composites reinforced with discontinuous phases (e.g., whiskers), the parameters contributing to the increased fracture toughness are described in terms of crack bridging by the reinforcement. A crack bridging model is discussed which is found to accurately predict the observed toughening response in SiC whisker-reinforced ceramics. While limited to these two toughening mechanisms, the paper will provide particular insight into the importance of microstructural control in achieving the desired toughening effects.

Transformation Toughened Ceramics

Marshall et al. have analyzed the energy change ΔG^T associated with the stress-induced martensitic transformation in the region adjacent to a propagating crack [23]. Using their results and recalling that $K = [GE/(1 - \nu^2)]^{1/2}$, the transformation-toughening contribution to the materials fracture resistance is:

$$\Delta K^T = \left[2E^c V^T \epsilon^T \sigma_c^T r_T/(1 - \nu^2)\right]^{1/2}. \tag{1}$$

The term, ϵ^T, is the volumetric transformation strain (a value of 4.5% is utilized), V^T is the fraction of tetragonal phase which transforms, E^c is the Young's modulus of the composite, ν is Poisson's ratio, and r_T is the width of the transformed zone [3c],[4]. As seen in Eq. (1), the transformation toughening contribution increases as the transformation zone size and amount of tetragonal phase which transforms increases. Note that r_T is proportional to $(K_I/\sigma_c^T)^2$ where K_I is the applied stress intensity. Thus, ΔK^T increases as σ_c^T decreases.

At this point, the ability to transform the tetragonal phase at the temperature of interest must be addressed. The tetragonal phase of zirconia is in equilibrium with the monoclinic phase at the temperature T_o where the chemical free energy change associated with the transformation is zero. Certain solutes (e.g., yttria, ceria) lower T_o and, thus, increase the stability of the tetragonal phase. The tetragonal phase can be present at temperatures below T_o when it is embedded in a matrix. The embedded tetragonal phase is prevented from transforming due to the resistance of the surrounding matrix to the required transformation volume expansion. The embedded tetragonal phase then does not begin to transform until the temperature T is decreased to the martensite start temperature, M_s. Decreasing T_o by solute additions will also decrease the M_s temperature of the embedded zirconia grain. At temperatures, T, between M_s and T_o, additional energy must be supplied to transform the metastable tetragonal phase. The energy required is:

$$\Delta U^T = \Delta S\left(M_s - T\right), \tag{2}$$

where ΔS is the transformation entropy change which has a value of -3.2 J/mol K.[24]

External work obtained by the application of a stress can be used to supply the additional energy required to transform the tetragonal phase. To promote the volume expansive martensitic transformation, an applied tensile stress $\geq \sigma_c^T$, the critical transformation stress, is required. When the resultant strain is primarily dilatational in nature, the external work required is:

$$W = \sigma_c^T e^T/2, \tag{3}$$

where ϵ^T is the dilatational component of the transformation strain. Combining Eqs. (2) and (3), the relationship between the transformation stress and the martensite start temperature is obtained:

$$\sigma_c^T = 2\Delta S(M_s - T)/e^T, \tag{4}$$

which reveals that σ_c^T will decrease as the material's M_s temperature is increased towards T, the test temperature (or T is decreased to M_s). Equation (4) can then be included in the zone size solutions [3c],[4] to determine the influence of the metastability of the tetragonal phase for test temperatures between T_o and M_s:

$$r_T = B\left(K_I/\sigma_c^T\right)^2 = B\left[K_I e^T/2\Delta S\left(M_s - T\right)\right]^2, \tag{5}$$

where B is $[3^{1/2}(1 + \nu)^2/12\pi]$ [3c],[4]. Equation 5 illustrates that the transformation zone around a crack in a given material will increase as the temperature of crack propagation T is decreased to M_s (or the material's M_s temperature is increased to T).

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Substituting Eqs. (4) and (5) into Eq. (1), the transformation-toughening contribution can be determined for $M_s<T<T_0$:

$$\Delta K^T = \left[C E^c V^T / \Delta S(M_s - T)\right]^{1/2} \epsilon^T K_I , \tag{6a}$$

where C equals $B/(1 - \nu^2)$. Thus, ΔK^T, at a desired test temperature, can be increased by altering the material's martensite start temperature, M_s.

Equation (6a) accounts for the increase in the transformation-toughening contribution for a selected applied stress intensity K_I. As K_I increases, the crack tip is shielded by the formation of the transformation zone which starts at the crack and extends outward as K_I increases. The peak or plateau transformation-toughening contribution (i.e, for long crack extensions) for a given material occurs when the crack tip stress intensity finally reaches the local fracture resistance of the material at the crack tip, K_m. This modification of Eq. (6a) can be expressed as:

$$\Delta K^T = \frac{K_m}{\left[C E^c V^T (\epsilon^T)^2 / \Delta S (M_s - T)\right]^{-1/2} - 1} , \tag{6b}$$

which again simply states that when $M_s < T$, the transformation toughening contribution will increase as M_s increases towards the test temperature or vice versa.

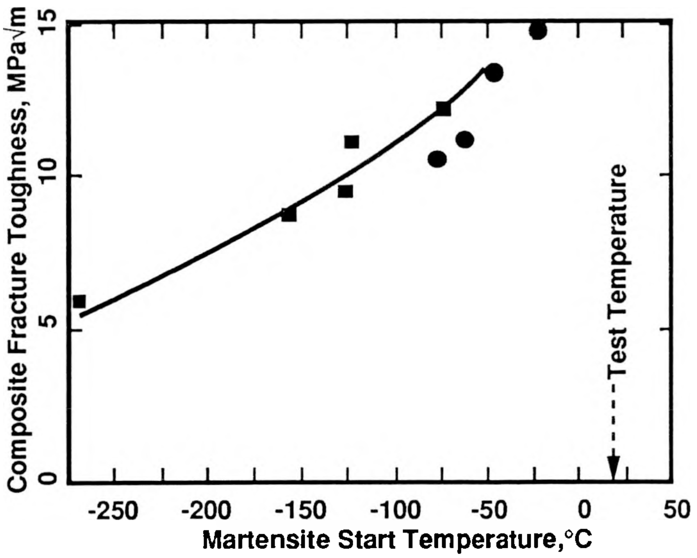

Fig. 1. The measured room temperature fracture toughness of zirconia-toughened alumina composites increases as the martensite start temperature, M_s, of the composite approaches the test temperature T when $M_s < T$. Data include alumina containing 40 vol % (■) ZrO_2 (12 mol % CeO_2) and polycrystalline tetragonal ZrO_2 (12 mol % CeO_2) (●). The short dashed vertical line indicates the test temperature.

This analysis shows that those factors which affect the M_s temperature of the zirconia grains embedded in a zirconia or other ceramic matrix can be used to change the transformation toughening contribution achieved at a desired temperature. As shown earlier, the M_s temperature of PSZ ceramics is raised with increase in the size of the tetragonal precipitates and, as predicted, the room-temperature fracture toughness increases with increase in M_s for $M_s < 20°C$ [24]. Similar behavior is observed in TZP ceramics where increasing grain size results in raising the M_s temperature and the room temperature toughness [25]. Claussen and Rühle showed that the M_s temperature of ZTC is also a function of the size of the tetragonal grains incorporated in an alumina matrix [5a]. The dependence of the fracture toughness of TZP and ZTA ceramics on the M_s temperature is illustrated in Fig. 1. In each case, the M_s temperature is a function of the size of the tetragonal zirconia grains and increases with increase in grain size.

If the solute (e.g., Y_2O_3, CeO_2) content is increased, the toughness at $T > M_s$ would be decreased as the tetragonal phase has greater stability (i.e., M_s will decrease as T_o decreases as mentioned earlier). However, the grain size dependence of the M_s temperature is, in part, also a result of local tensile stresses generated by the thermal expansion anisotropy of TZP and mismatch in ZTC ceramics. As seen in studies by Schubert and coworkers, the crystallographic thermal expansion coefficients of tetragonal zirconia are a function of solute (i.e., yttria) content [26]. Thus, increasing the solute content not only decreases T_o, which in effect would decrease M_s, but also can decrease the local residual tensile stresses which would diminish the grain size effect on the M_s temperature. Both these factors would increase the resistance of the tetragonal phase to transforming and lower the transformation-toughening contribution at a given temperature. This is reflected in the behavior of alumina-20 vol % zirconia composites where the fracture toughness at selected temperatures decreases with increasing amounts of yttria solute in the zirconia [5b].

As noted in Eq. (6), the transformation-toughening contribution will increase as the amount of tetragonal phase, which is included, transforms. Simply increasing the amount of tetragonal phase added is beneficial only if a greater fraction also transforms [3c],[4],[5b]. Additional studies indicate that the ability to transform tetragonal zirconia grains embedded in alumina can also be influenced by the volume content of zirconia added. This also is seen to result from the effects of the tensile components of the residual stresses introduced due to the thermal expansion mismatch between the alumina and zirconia [5b]. The effect is such that the tetragonal zirconia grains will experience greater residual tensile stresses due to the approach of neighboring zirconia grains as the zirconia content is increased. At each selected solute content, the tetragonal grains will be transformed during post fabrication cooling when the zirconia content is raised above a critical level. Increasing solute content allows greater amounts of zirconia to be added and retained in the tetragonal phase. Therefore, the toughness reaches a maximum with increase in zirconia and then decreases when the zirconia content exceeds the critical amount [5b]. The result is that incorporating increasing amounts of tetragonal phase into alumina requires that the solute content be increased and/or the zirconia grain size be decreased to retain the tetragonal phase. However, while the M_s temperature is decreased and the retention of the tetragonal phase is increased by solute additions and grain size reductions, the toughness achieved (e.g., at room temperature) is also affected, Fig. 2. Simply increasing the solute content to be able to include and retain more tetragonal zirconia in alumina may not result in greater toughness if too much solute is added (or the grain size is too small). Obviously, the optimization of the transformation-toughening contribution requires careful control of the zirconia grain size and solute content for each level of zirconia added to the ceramic.

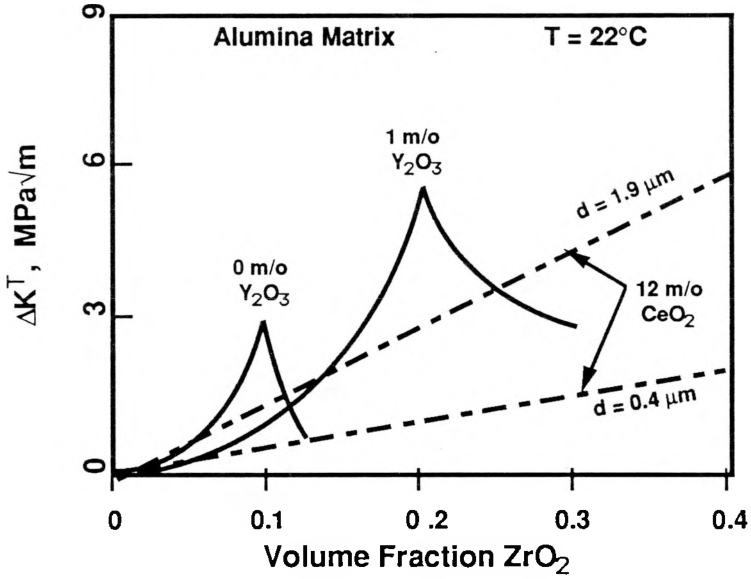

Fig. 2. The transformation toughening achieved in zirconia-toughened ceramics is dependent upon the volume fraction of zirconia added, the solute content of the zirconia, and the zirconia grain size, d. The addition of solute (e.g., yttria) allows more zirconia to be added and retained as the tetragonal phase and increases the peak toughness achieved (e.g., in zirconia with d equal to 0.6 to 0.8 μm) with 0 vs 1 mol % yttria. This affects the rate of increase in toughness with zirconia content as the tetragonal phase is more difficult to transform (e.g., 0 vs 1 mol % yttria solute content). Increasing the zirconia grain size also enhances the toughening contribution (e.g., d equal to 0.4 μm vs 1.9 μm) in zirconia containing 12 mol % ceria.

The results of Eq. (6) also point out that the toughness achieved in any system of a given composition and microstructure will be dependent on the test temperature as well as the M_s temperature of that material. That is, in fact, observed in PSZ [24], TZP, and ZTC ceramics, Fig. 3. If high toughness is desired over a selected temperature range, both the solute type and amount and the microstructure must be controlled to adjust the M_s temperature of the material. These and earlier findings show that one must carefully consider the effects of composition and microstructure in the development of transformation-toughened ceramic systems for various applications.

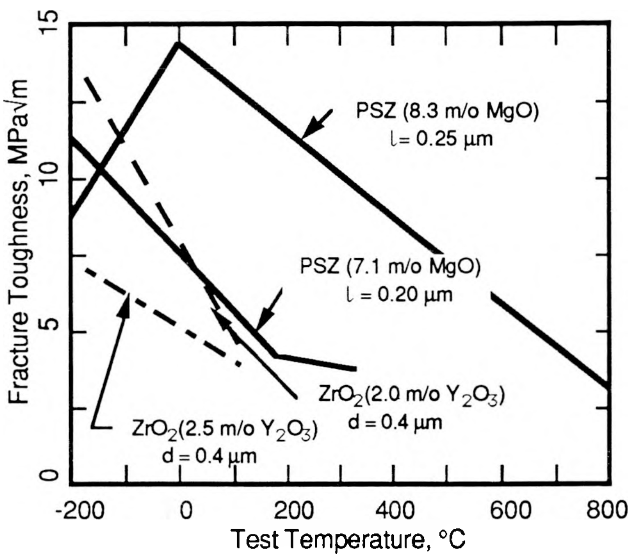

Fig. 3. The fracture toughness of transformation-toughened ceramics is markedly affected by the test temperature. Decreasing the martensite start temperature M_s by either increasing addition of solute (e.g., yttria in TZP of constant grain size, d) or decreasing the size of the tetragonal phase (e.g., tetragonal precipitate length l in PSZ) decreases the toughness achieved at a given temperature (e.g., room temperature). A maximum in toughness is obtained when the test temperature is decreased and approaches the M_s temperature of each material.

Toughening by Discontinuous Reinforcements

Crack bridging by various brittle, nontransforming reinforcing phases can result in significant toughening of a ceramic. Indeed, the reinforcing phase can consist of (1) a second phase which is added prior to or formed during processing or (2) matrix grains of appropriate size or shape. Several bridging processes can be introduced: elastic bridging with or without interfacial debonding between the matrix and the reinforcement, frictional bridging along the debonded interface, and pullout of the debonded reinforcement. The analysis of the toughening contribution is briefly described and the reader is referred to earlier studies for more details [3b],[6],[7],[10],[12]. The bridging contribution to the toughness is:

$$\Delta K^{cb} = K^c - K^m = (E\Delta J)^{1/2}, \tag{7}$$

where K^c is the overall toughness of the composite, K^m is the matrix toughness, and the term ΔJ corresponds to the energy consumption due to the bridging process. The energy consumed by bridging processes is a function of the bridging stress, T_u, the crack opening displacement, u profile:

$$\Delta J = \int_0^{u_{max}} T_u du , \tag{8}$$

associated with each bridging process noted above where u_{max} is the maximum displacement at the end of each type of bridging zone [6]. Unless the tensile strength of the reinforcement is extremely high (i.e., ~E/10 for reinforcement of polycrystalline matrices), elastic bridging in the absence of interfacial debonding is not seen as making a substantial contribution. On the other hand, elastic bridging of partially debonded reinforcements, frictional bridging, and pullout can impart substantial toughening effects.

For these bridging processes, various material parameters will control the toughness achieved as shown in the following results. The energy consumption associated with elastic bridging associated with partial interfacial debonding is:

$$\Delta J^{eb} = A^{el} \left(\sigma_f^l\right)^2 l_{db} / 2E^l , \tag{9}$$

where A^{el} is the areal fraction of bridging reinforcement on the crack plane, σ_f and E^l are the tensile strength and the Young's modulus, respectively, of the reinforcement, and l_{db} is the debonded length of the interface. Using the results of Budiansky et al. [27], the debonded length can be defined as ($l_{db} = \gamma^l/6\gamma^i$) where γ^l and γ^i represent the reinforcement and interfacial fracture energies, respectively. One can envision elastic bridging with debonded interfaces occurring (1) if radial tensile stresses were imposed on the interface due to a smaller matrix thermal expansion coefficient, α_m, than that of the reinforcement, α_r, and/or (2) a reinforcement with a high Poisson's ratio. In either case, the reinforcement contracts away from the matrix either during post-fabrication cooling or under tensile load.

When frictional forces are imposed at the debonded interface, the energy consumed becomes:

$$\Delta J^{fb} = A^{fb} r \left(\sigma_f^l\right)^3 / 6\tau_i E^l = A^{fb} \left(\sigma_f^l\right)^2 l_{db} / 3E^l , \tag{10}$$

where the superscripts signify frictional bridging parameters, and τ_i is the interfacial frictional shear stress. When pullout occurs, the energy consumption becomes:

$$\Delta J^{po} = A^{po} \tau_i l_{po}^2 / r , \tag{11}$$

where the superscripts denote the parameters for pullout (i.e, l_{po} is the length of the pullout). Note that pullout can, but does not necessarily, accompany frictional bridging; this will be dictated by failure of the reinforcement at a position at some distance from the matrix crack plane (i.e., $l_{po} > 0$).

Thus the properties and characteristics of the reinforcement and the interface play a major role in the toughness achieved by each of these bridging processes. For example, the incorporation of strong whiskers (e.g., studies indicate that SiC whiskers can exhibit strengths of up to 16 GPa) [28] is necessary to achieve significant toughening effects. Factors such as the topography of the reinforcement surface also can be a factor. If the reinforcement exhibits a corrugated/stepped surface, this can influence its strength as surface steps can act as stress concentrations to lower the strength. Also, when the matrix conforms to this corrugated shape, it forms an interlocking interface and frictional displacement between the reinforcement and matrix is inhibited. Such features will limit the length of the debonded interface and frictional sliding of the reinforcement. Furthermore, the frictional shear resistance and debonding will be influenced by the mismatch in properties of the reinforcement and matrix and also the chemical bonding at the interface. For example, thermal expansion differences between the matrix, α_m, and reinforcement, α_r, can impose radial stresses on the interface as noted earlier. Tensile radial stresses acting on the interface tend to promote debonding and decrease the interfacial frictional shear resistance. On the other hand, compressive radial stresses generated when $\alpha_m > \alpha_r$ would increase the interfacial shear resistance. If the interface consists of a weak bond or phase, radial compressive stress would limit the amount of debonding, but not necessarily prevent it. Keeping these factors in mind, the results of Eqs. (9)-(11) indicate that the design of reinforced ceramics can be achieved by accessing the important material properties and characteristics.

There are numerous examples of the contribution of discontinuous brittle reinforcements to the fracture toughness of ceramics including second-phase whiskers [6],[10],[17] and platelets [10],[29] and both elongated [7], plate-like [30] and large [7] matrix grains. Studies of cracks in such materials reveal that, within the wake of the crack tip, the reinforcing entities do bridge the crack. The following sections will only briefly describe some of the toughening effects that are achieved with emphasis on role of those material parameters contained in the above analyses.

Whisker Reinforcement

The experimental results obtained with a selected SiC whisker reveal that the parameters contained in the models for crack bridging, indeed, exert a strong influence on the crack bridging toughening as seen in Fig. 4.[6] First, the whisker bridging toughening contributions from frictional bridging and pullout bridging increase with volume/areal content of the reinforcing phase as predicted. Second, the toughening contribution also increases as the ratio of the composite's Young's modulus to that of the whisker increases. This is best illustrated by the rise in ΔK^{wr} with increase in E^c at a given whisker content. For the examples here, E^c values were obtained by rule of mixtures [$E^c = E^m(1 - V_f) + E^w V_f$]; thus, at a constant volume fraction of whiskers, E^c increases in the order from glass ($E^m = 80$ GPa) to mullite ($E^m = 210$ GPa) to alumina ($E^m = 400$ GPa) vs SiC ($E^w = 500$ GPa).

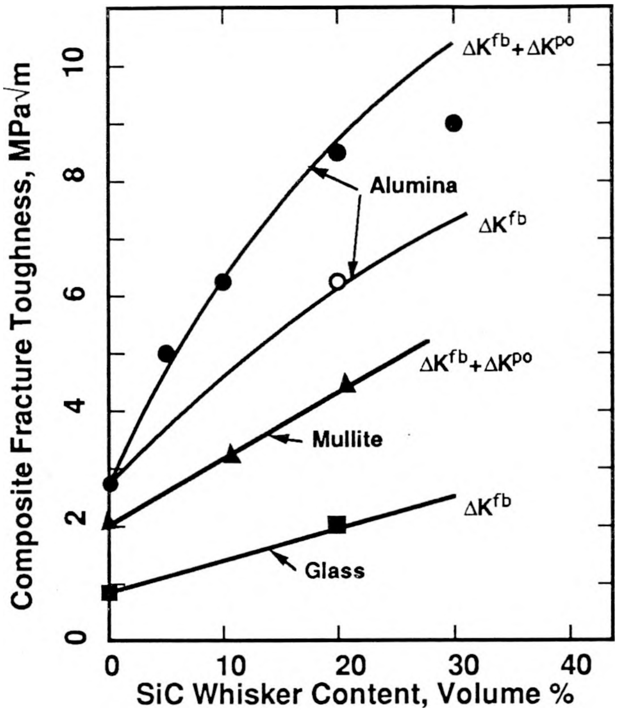

Fig. 4. The toughening contributions obtained by crack bridging processes yield substantially increased fracture toughness. The curves represent the behavior predicted for frictional bridging ΔK^{fb} and the combined effects of frictional bridging plus pullout ΔK^{po} for alumina, mullite, and glass matrix composites. The filled symbols indicate experimental data obtained with as-received 0.8 μm diam SiC whiskers; the open circle is that for an alumina composite made with the same whiskers in a preoxidized condition [from "Microstructural Design of Toughened Ceramics," P. F. Becher, *J. Am. Ceram. Soc.* 74(2), 255-69 (1991); reprinted with permission of the American Ceramic Society].

Additional observations also show that the frictional bridging toughening contribution, ΔK^{fb}, becomes greater as $r^{1/2}$ (the whisker radius) increases [6],[10]. For example, the toughness of alumina composites containing 20 vol % SiC whiskers, increased from $\approx$6.5 to $\approx$9 to $\approx$12 MPa $m^{1/2}$ when the mean diameter of the SiC whiskers increased from 0.4 to 0.75 to 1-1.5 microns, respectively. These results are obtained with whiskers which have smooth surfaces. Our own studies have shown that larger diameter whiskers which have large surface steps do not result in substantial toughening (e.g., toughness values of only 6 to 7 MPa $m^{1/2}$ are obtained for alumina with additions of ~20 vol % ~1 μm diam, corrugated SiC whiskers).

From the toughening model, we also expect the toughness to increase as the matrix-whisker interface fracture energy (strength) decreases with respect to that of the whisker (e.g., $l_{db} \propto \gamma^w/\gamma^i$). While values of the ratio of the whisker to interface fracture energy (γ^w/γ^i) are not available, there are two observations which support the predicted behavior. First, significant toughening is observed only when whisker-matrix interfacial debonding and crack bridging by the whiskers are observed. Second, the toughness of a particular system (e.g., alumina-SiC whiskers) is greatest when whisker pullout is evidenced by the protrusion of whiskers above the fracture surface. This is noted in the samples fabricated with as-received vs preoxidized whiskers where the toughness, Fig. 4, is diminished by preoxidation of the whiskers. These findings indicate that the pullout contribution was affected. Also, noted in Fig. 4, the predicted toughening curves for mullite and glass matrices suggest that the pullout contribution is small relative to that which could be achieved in alumina (see Ref. 6). This is substantiated by observations of the crack surfaces for the three different matrix composites where the length of the SiC whiskers protruding above the crack plane is much shorter for the mullite matrix and negligible for the glass matrix composites.

Thus, it appears that crack bridging by the SiC whiskers can be influenced by pretreatment, oxidation, or reduction of the SiC whisker surfaces [10]. This could result from differences in the debonding and/or frictional shear resistance of the interfaces due to changes in the interface structure. However, subsequent high resolution electron microscopy studies of the interfaces of SiC whisker-reinforced alumina composites fabricated from both as-received and preoxidized whiskers reveal that factors such as the interfacial amorphous phase, which is generally present, are not substantially altered by such pretreatments [31]. Thus, one can only speculate as to those parameters which are affected by such pretreatments. One should keep in mind that the strength of the whisker and the nature of the chemical bond of the interfaces could both be affected by such pretreatments of the whiskers. Additional studies are needed to elucidate these factors.

The key points illustrated by these results for the toughening contribution by crack bridging processes in whisker-reinforced ceramics are as follows. First, the toughening contribution is enhanced through the use of very strong whiskers which have a smooth surface topography and can be increased by increasing the whisker diameter. Second, the toughness increases with increase in whisker content. Third, enhanced toughness is achieved with increase in the Young's modulus of the matrix relative to that of the reinforcement. Fourth, interfacial debonding is necessary to achieve significant bridging effects.

Role of Matrix Microstructure

As illustrated by several authors, the matrix grain size, especially in noncubic ceramics, can be a factor in the toughness achieved as long as intergranular debonding occurs [7a],[7b],[7c]. This grain size effect is well-documented in alumina ceramics and is also attributed to crack bridging by the matrix grains. The resulting crack bridging and, hence, the toughness increases with increase in the matrix grain size. The toughening contribution due to matrix grain bridging can be obtained by analogy with that obtained above for pullout of the whisker reinforcement. The bridging stress supported by matrix grain bridges here is taken as the product of the frictional stress required to pull out each bridging grain times the areal fraction of bridging grains (e.g., $A^{gb}\tau_{gb}$). In order to obtain the crack opening displacement at the end of the grain bridging zone, the maximum crack opening displacement is equated to that required to completely pull out the bridging grains. For simplicity, it is assumed that the pullout length is equal to half the grain diameter. The resultant incremental increase in the energy consumption due to pullout of bridging matrix grains is then:

$$\Delta K^{gb} = \left[A^{gb}\tau_{gb}E^{c}(d/2)\right]^{1/2}, \tag{12}$$

where d is the matrix grain size. This yields a grain bridging toughening contribution consistent with experimental observations for alumina ceramics [6],[7a],[7b],[7c]. Therefore, a degree of toughening can be achieved, at least in noncubic ceramics, by controlling the grain size. On the other hand, it is recognized that high fracture strengths generally require grain size refinement in materials such as alumina. Thus, the microstructure must be tailored with respect to the grain size required to meet the particular strength and toughness requirements in these monolithic ceramics. As seen from the earlier discussions, this can be aided by including either transformation toughening or reinforcement processes.

Toughening by Matrix Grain Morphology

Crack bridging phenomena and toughening effects, which are very comparable to those observed in whisker-reinforced ceramics, are also found in ceramics containing other reinforcing phase geometries. Reinforcement of Si_3N_4 ceramics by the *in situ* growth of elongated or whisker-like β-grains has been observed with toughness values of up to at least 10 MPa $m^{1/2}$ [7d],[7e],[7f],[7g] as compared to values of ≤ 5 MPa $m^{1/2}$ for Si_3N_4 ceramics with fine, equiaxed microstructures. Observations of the crack paths in these self-reinforced Si_3N_4 ceramics reveal that crack bridging by and pullout of these grains is associated with the improvement in fracture toughness [7e]. Tajima et al. also showed that the toughening contribution of the bridging elongated grains increased as the volume fraction of the elongated grains increased [7f]. Further studies show that the toughening contribution due to bridging by these elongated grains increases with increase in the square root of the diameter of the elongated grains [7g]. This is consistent with the bridging behavior predicted by Eqs. (10) and (11).

Therefore, it is apparent that considerable toughening can be achieved by tailoring the matrix grain shape during processing with the additional requirement that debonding must occur at the interface between the elongated grains and the remaining matrix grains. Unlike the behavior of toughening by bridging of large grains in alumina, toughening by bridging elongated grains can also yield materials with high fracture strengths in silicon nitride ceramics. This toughening-strength response for ceramics with elongated grain structures, then, offers substantial promise for the development of ceramics with excellent mechanical performance for high-stress applications.

Coupled Toughening Approaches

An area with exceptional potential for developing toughened ceramics with excellent mechanical properties involves the combination of toughening processes. These can include any of the approaches discussed here or additional processes such as continuous fiber reinforcement among others. Early studies revealed that whisker reinforcement can be combined with transformation toughening achieved by adding zirconia particles [32]. In this case, the combined effects of transformation toughening and whisker reinforcement resulted in fracture toughness values which were greater than the sum of the contribution from each individual process. Over five-fold increases in toughness were achieved in mullite with the addition of 20 vol % each of zirconia particles and SiC whiskers. Under conditions where the zirconia particles were already transformed, toughening by microcracking associated with monoclinic zirconia combined additively with whisker reinforcement to yield two- to three-fold increases in the fracture resistance.

One can also combine toughening by various crack bridging processes. This is seen in the coupling of toughening achieved by reinforcing second phases with tailoring of the matrix microstructure. It was shown that the fracture resistance of SiC whisker-reinforced aluminas can be enhanced by increasing the alumina matrix grain size as well as by increasing the whisker content [6]. The combination of toughening by elongated matrix grains with that from reinforcing phases (i.e., whiskers) has also been employed with some success [33]. The results achieved by combining these particular toughening processes are complicated by the fact that the addition of reinforcing phases tends to inhibit the growth of either large grains (e.g., in alumina) or the elongated matrix grains (e.g., in silicon nitride) [6]. However, it is possible to enhance the fracture toughness of such composites by tailoring the matrix grain size and shape, as well as by incorporating reinforcing second phases.

Conclusions

The fracture resistance of ceramic systems can be substantially improved by a number of different approaches, and these can be combined to obtain additional toughening effects. The successful application of these various approaches demands that attention be paid to the influence of microstructure and the chemical composition on the toughening response and how the response may be modified by the temperatures that the ceramic is exposed to. The text here has obviously not exhausted all the possible toughening mechanisms but has chosen instead to examine those of transformation toughening, whisker and related reinforcement approaches such as matrix grain size and shape, and the combined effects of these.

The behavior of transformation toughening zirconias are quite well described by the existing models. Keeping account of the influence of the tetragonal phase particle/grain size and of the solute content, one can realistically design toughened zirconias for a design application, especially with respect to use temperature. Further insight into the alloying behavior of these systems (e.g., what solute characteristics influence the strength of its stabilizing effect) would be of great benefit. In the case of other zirconia toughened ceramics, one can draw on the knowledge of the transformation toughened zirconias. However, one must account for the effects of the mismatch in matrix vs zirconia particle properties and resultant local stresses on the ability to transform the tetragonal phase particles. One also needs to understand how these stresses change with increase in both solute content and volume fraction of zirconia included in the ceramic. We have guidelines now for the influence of zirconia grain size in TZP or particle size in ZTC can be used to enhance both the transformability of the tetragonal phase and the associated transformation toughening contribution. These factors and the matrix microstructure effects are seen as root causes in the variability in the fracture toughness often observed in zirconia and zirconia toughened ceramics.

Progress in the area of reinforced ceramics is providing a wealth of new insights into the optimization of the toughening of ceramic systems. The advances in the theoretical description of the toughening response in whisker- and fiber-reinforced ceramics provide details of how to begin to design tougher materials and directions where more insight is needed (e.g., interfacial property-structure relationships). These two systems, whisker- vs fiber-reinforced, cover a range of systems—those based on very weak interfaces to those utilizing quite strong interfaces and those in between which only strengthens the need to be able to control interfacial phenomena and their effects on fracture resistance. In addition, the models for whisker reinforcement point to a need to develop techniques to synthesize whiskers where size and strength can be altered in a controlled manner and strength maximized.

The use of microstructural control to toughen ceramics offers considerable potential, and along with the reinforcement approaches suggests other avenues for toughening. In materials like the toughened silicon nitrides with elongated grain structures, it is important to determine what and how grain and interfacial/grain boundary properties can be modified to optimize the toughness. This area and the ability to combine toughening processes are exciting new fields for exploration and exploitation in the design of ceramic materials.

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